

NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE 3927

PRELIMINARY INVESTIGATION OF THE EFFECT OF SURFACE
TREATMENT ON THE STRENGTH OF A TITANIUM CARBIDE -
30 PERCENT NICKEL BASE CERMET

By Leonard Robins and Edward M. Grala

Lewis Flight Propulsion Laboratory
Cleveland, Ohio



Washington

February 1957

FOR REFERENCE

LIBRARY COPY

MAR 1 1957

LANGLEY AERONAUTICAL LABORATORY
LIBRARY, NACA
LANGLEY FIELD, VIRGINIA

NOT TO BE TAKEN FROM THIS ROOM

NATIONAL ADVISORY COMMITTEE FOR AERONAUTICS

TECHNICAL NOTE 3927

PRELIMINARY INVESTIGATION OF THE EFFECT OF SURFACE TREATMENT

ON THE STRENGTH OF A TITANIUM CARBIDE - 30 PERCENT

NICKEL BASE CERMET

By Leonard Robins and Edward M. Grala

SUMMARY

The effect of various surface treatments on the room-temperature modulus of rupture and impact strength of a nickel-bonded titanium carbide cermet were investigated. The average strengths for the treatments varied from about 200,000 to 80,000 pounds per square inch in modulus of rupture, and about 3.5 to 1 inch-pound in impact resistance. The strengths of lapped, grit-blasted, diamond-ground, or vapor-blasted specimens were not significantly different. The most serious losses of strength occurred after oxidation (at 1600° F for 100 hr), surface roughening by acid attack, and severe grinding with a 60-grit silicon carbide abrasive wheel. The modulus-of-rupture strength of oxidized specimens was improved after grit blasting or regrinding with a diamond abrasive wheel. The magnitude of the changes in impact and modulus-of-rupture strengths for some surface treatments were quite different.

INTRODUCTION

The currently poor reliability of cermet turbine blades prohibits their use in jet engines. Since cermets are relatively brittle, small surface imperfections can be expected to result in a large loss of strength and impact resistance and, consequently, to influence the reliability. These surface imperfections may originate during fabrication. In addition, a cermet turbine blade is exposed to a number of conditions during operation (e.g., erosion, abrasion, corrosion, and oxidation) which can further alter the surface. Theoretical and experimental investigations of the effect of surface condition on the strength of several brittle materials have shown that small surface defects can radically affect strength properties (e.g., refs. 1 and 2); however, no such study has been reported for cermets.

In order to provide information on the effect of surface treatment on the strength of cermets, an exploratory investigation has been made to determine the room-temperature modulus-of-rupture and impact strengths of titanium carbide - nickel base cermet specimens which received various surface treatments. The types of surface treatment employed were grinding, lapping, blast cleaning, acid roughening, and oxidizing. Some oxidized specimens were refinished by grinding or grit blasting.

Several of the treatments used in this study were selected because they are pertinent to current experimentation with cermet turbine blades. Diamond grinding is used to grind turbine blade roots; vapor blasting is often used to give sintered cermet turbine blades a uniform finish; lapping is sometimes used to improve the concentricity and fit of mating components (e.g., at the root section of cermet turbine blades); and the blades oxidize during operation. On the other hand, to exaggerate some of these effects, other treatments selected were as follows: using a large vertical feed per pass to obtain a badly ground surface and chattermarks with silicon carbide abrasive; acid roughening; and steel-grit blasting.

MATERIALS AND PROCEDURES

Specimens

The cermet specimens were produced commercially by powder-metallurgy techniques from a single batch of carbide and nickel powders. The composition, designated K152B, is nominally 62 percent titanium carbide, 8 percent complex carbide of niobium, tantalum, and titanium, and 30 percent nickel by weight. The structure consists of a mixture of angular carbide particles, metal binder (a nickel-rich solid solution), and perhaps graphite. Representative microstructures are shown in figure 1; the carbides appear dark gray, and the binder phase appears white. It is believed that some of the black area is graphite, while the remainder is porosity and holes which result from "pull-outs" during cutting, grinding, and polishing.

Twelve "as-received" bars were radiographically inspected and, since no internal defects were observed, the remaining bars were presumed to be sound and were not inspected.

Surface Treatment

The surface treatments are described in table I. The bars listed in column A were received rough ground to a 0.23-inch square. Prior to the surface treatments shown these bars were ground, using the procedure listed under treatment I, to the pretreatment dimensions shown on

the table. The bars in column B were received ground to a 0.1875-inch square by the procedure listed under treatment II and then surface treated. Except where noted in the remarks, all final cross sections were within ± 0.0005 inch of 0.1875-inch square. The length was 1.50 inch. In those cases where the surfaces were prepared by grinding or lapping, the abrading was done parallel to the longitudinal axis of the specimens.

Characterization of Surfaces

A description of the test specimens after surface treatment is presented in table II. Except for the penetrant-oil examination, one specimen per surface treatment was used in each of the following examinations:

Penetrant oil. - The surfaces of all specimens were examined after treating with a post-emulsifier penetrant oil.

Macroappearance. - A photograph of the magnified surfaces of fractured specimens is shown in figure 2. Since luster should, to some extent, depend on the surface roughness of the specimens (except in the case where the specimen was oxidized or etched), figure 2 has been arranged, from left to right, in order of visually apparent decreasing luster.

Microstructure. - Photomicrographs of the edges of transverse sections are shown in figure 1. The sections were cut from fractured test specimens.

Hardness. - Hardness measurements were made on the treated surface of fractured specimens using the A scale of a standard Rockwell tester. The hardness value was the average of four or more measurements.

Roughness. - A profilometer (surface analyzer) was used to indicate the surface roughness of the test specimens. The instrument measures, in microinches, the root-mean-square deviation from the mean surface. Except for the diamond-ground specimens, the differences in roughness values for traverses made parallel and perpendicular to the longitudinal direction in the specimen were very small and within the experimental scatter. The exception is noted in table II.

Evaluation of Strength

A minimum of two specimens was evaluated for each surface treatment in the following room-temperature strength tests:

Modulus of rupture. - A 120,000-pound-capacity testing machine was used for modulus-of-rupture evaluations. The specimens were supported

0.25 inch from each end (distance between supports is 1 in.) by hardened steel rods and broken by loading with an opposed, centrally located, hardened steel rod. The rate of loading was 22,000 pounds per square inch per minute. The modulus of rupture is the maximum tensile stress developed at a surface of the specimens and is calculated from the following equation:

$$\text{Modulus of rupture} = 1.5 \frac{(\text{transverse breaking load})(\text{span length})}{(\text{specimen width})(\text{specimen thickness})^2}$$

Impact strength. - A 25.5-inch-pound-capacity Izod pendulum testing machine was used to determine the unnotched impact strength of the specimens. A detailed description of the equipment and evaluation procedure is given in reference 3.

RESULTS AND DISCUSSION

Modulus-of-rupture and impact-strength data are given in table III, and the average strength values are graphically shown in figure 3. The results are as follows:

(1) There is little difference among the modulus-of-rupture and impact strengths after lapping, diamond-abrasive-grinding, vapor-blasting, or grit-blasting treatments. The strengths after these treatments are the highest obtained in this study.

(2) Despite the fact that the lapped specimens were equal or superior to the others on the basis of all surface inspections (table II), their strengths were not unusually high and, in fact, they may have suffered a small loss in modulus of rupture. There is no explanation for this behavior.

(3) Significant differences were revealed by the inspections of the diamond-ground and vapor- or grit-blasted surfaces, particularly in the microstructure (figs. 1(a), (b), (e), and (f)) and the surface roughness (table II). As stated previously, there is no significant difference between the strengths after treatment. Possible reasons for this are:

(a) The grinding was in a direction parallel to the applied stress. Had the direction of grinding been normal to the stress, significant differences might have been revealed.

(b) The vapor- or grit-blasting treatments resulted in some rounding of the edges of the test specimens. This rounding may have offset the expected loss of the strength because of roughening.

(4) As expected on the basis of the surface inspections, oxidizing or acid roughening both result in a low modulus of rupture and the lowest impact strengths.

(5) Refinishing oxidized specimens by diamond-abrasive grinding or steel-grit blasting results in an appreciable recovery of the modulus of rupture. This result was achieved with a negligible reduction of the cross-sectional area. No impact tests were made on the refinished specimens.

(6) Severe grinding (with silicon carbide abrasive), which caused chatter marks and considerable surface scratching, results in the lowest modulus of rupture for any of the treatments, while the impact strength was about the same as the high values of those in result (1).

While only preliminary conclusions concerning the effects of the various treatments can be drawn from the data, the results indicate that the strength of this titanium carbide - nickel base cermet can be varied over a range of from about 200,000 to 80,000 psi in a modulus-of-rupture test, and 3.5 to 1 inch-pounds in an impact test, by surface treating alone. It is interesting to note that because of the different natures of the modulus-of-rupture and impact tests, the surface treatments are rated in a different order by each test.

The low strength of the oxidized specimens is of special importance since cermet turbine blades are exposed to oxidation during service in jet engines; this result will be considered further. As stated in table II, no microstructural changes appear to occur after 100 hours at 1600° F in the zone below the oxidized layers. This is in agreement with reference 4, which states that no age hardening was observed and the properties are unchanged by heat treatment. Therefore, the low modulus-of-rupture and impact strength must be due to the oxidation. This strength loss due to oxidation may explain the steep stress-to-rupture curves for this material (ref. 5, pp. 1, 3, and 76). The preceding hypothesis differs from a tentative theory, based on a solution-reprecipitation mechanism resulting in coalescence of the carbide particles, which has been advanced to explain the relatively steep slope (ref. 5, pp. 1-23). The detrimental effect of oxidation on the strength of metal-bonded titanium carbide cermets has, however, also been noted by others (ref. 6, p. 988). In this reference, on the basis of a few tests, the decrease in modulus of rupture after oxidation was demonstrated to be proportional to the thickness increase.

Additions to improve the oxidation resistance of nickel-bonded titanium carbide cermets that have been reported include a solid-solution carbide of niobium, tantalum, and titanium (ref. 6); chromium in solution with the binder metal (ref. 7); chromium carbide (ref. 4); and silicon carbide (ref. 8). The first addition (ref. 6) is the one

incorporated in the material reported herein. This addition is not entirely satisfactory with regard to retaining the original strength on the basis of the strength loss after exposing this material to oxidation at 1600° F for 100 hours (result (4)). It is possible that the other additions (refs. 4, 7, and 8) produce more effective protection against oxidation. The data also suggest that the vapor- or grit-blasting processes may offer a simple and inexpensive means of recovering the properties of cermets whose surfaces have been damaged by oxidation (see result (5)).

SUMMARY OF RESULTS

The room-temperature modulus-of-rupture and impact strength were determined for a 70 percent carbide - 30 percent metal titanium carbide - nickel base cermet after several different surface treatments. Some surface treatments markedly altered the strength properties. Principal losses in strength occurred after the as-ground specimens were given one of the following surface treatments: Oxidation at 1600° F for 100 hours; roughening with aqua regia; and severe grinding with a silicon carbide abrasive wheel. The modulus of rupture of oxidized specimens increased after refinishing the surface by grit blasting or grinding with a diamond abrasive wheel. Lapping with diamond powder, steel-grit blasting, and vapor blasting had little or no effect on the as-ground strength.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, December 11, 1956

REFERENCES

1. Griffith, A. A.: The Phenomena of Rupture and Flow in Solids. Phil. Trans. Roy. Soc. (London), ser. A, vol. 221, Mar. 1921, pp. 163-198.
2. Griffith, A. A.: The Theory of Rupture. Proc. First Int. Cong. for Appl. Mech., Delft (Holland), 1924, pp. 55-63.
3. Probst, H. B., and McHenry, Howard T.: A Study of the "Toss Factor" in the Impact Testing of Cermets by the Izod Pendulum Test. NACA TN 3931, 1957.
4. Trent, E. M., Carter, A., and Bateman, J.: High Temperature Alloys Based on Titanium Carbide. Metallurgia, vol. 42, no. 250, Aug. 1950, pp. 111-115.

5. Shevlin, Thomas S., Newkirk, Herbert W., Stevens, E. George, and Greenhouse, Harold M.: Preliminary Microscopic Studies of Cermets at High Temperatures. WADC Tech. Rep. 54-33, pt. 2, Wright Air Dev. Center, Wright-Patterson Air Force Base, Feb. 1956. (Contract AF 33(038)-16911.)
6. Redmond, J. C., and Smith, E. N.: Cemented Titanium Carbide. Trans. A.I.M.M.E., Inst. Metals Div., vol. 185, Dec. 1949, pp. 987-993.
7. Schwarzkopf, P., and Kieffer, R.: Refractory Hard Metals. Macmillan Co., 1953, pp. 396-398.
8. Wilson, R. E., Coffin, L. B., and Tinklepaugh, J. R.: Metal and Self-Bonded Silicon Carbide. WADC Tech. Rep. 54-38, pt. 2, Wright Air Dev. Center, Wright-Patterson Air Force Base, Jan. 1955. (Contract AF 33(038)-16911.)

TABLE I. - PREPARATION OF SURFACE-TREATED SPECIMENS

Treatment A B		Number of specimens	Pretreatment cross section, in. sq	Surface treatment	Abrasive or reagent	Coolant	Surface speed, ft/min	Vertical feed per pass, in.	Remarks
I		4	0.23	220-Grit diamond grinding	220-Grit resinoid- bonded diamond wheel	Water and soluble oil	5000	0.0005- 0.0010	
	II	4	Pregrinding cross section unknown	100-Grit diamond grinding	100-Grit resinoid- bonded diamond wheel	Unknown	Unknown	Unknown	
III		8	0.190	Hand lapping	45 μ diamond paste; final 0.0005 in. removed with 6 μ diamond paste	None	-----	-----	
IV		4	0.23	Severe silicon carbide grinding	60-Grit vitreous- bonded silicon carbide wheel	Water and soluble oil	5000	0.002- 0.003	Resulted in "chatter- marks" on surface
V		4	0.190	Vapor blasting	40-80 Grit crushed silica sand- water spray (noz- zle pressure, 600 lb/sq in.)	Water	-----	-----	
VI		4	0.190	Grit blasting	0.03 to 0.04-in. angular steel particles (nozzle pressure, 1000 lb/sq in.)	Air	-----	-----	
VII	VII	4,4	0.1875	Oxidizing	Air at 1600° F for 100 hr	-----	-----	-----	Slight buildup of oxide at surface; final cross section of 0.190 in. sq
VIII		2	0.1875	Oxidizing and 220- grit diamond grinding	Air at 1600° F for 100 hr followed by treatment I	-----	-----	-----	
IX		2	0.1875	Oxidizing and grit blasting	Air at 1600° F for 100 hr followed by treatment VI	-----	-----	-----	
X		4	0.195	Acid roughening	Warm aqua regia for 2 hr	-----	-----	-----	^a Final dimensions were 0.165 in. sq

^aRate of surface removal was greater than expected.

TABLE II. - CHARACTERIZATION OF SURFACE-TREATED TITANIUM CARBIDE - NICKEL BASE CERMET (K152B)

Surface treatment	Results of surface examination				
	Penetrant oil	Macroappearance (see fig. 2)	Microstructure (see fig. 1)	Hardness, Rockwell A	Surface roughness, rms microin
220-Grit diamond ground	Fine pin-point "porosity" which may be the result of "pull-outs"; typical of ground surfaces for this material	Typical of ground surfaces	Numerous tiny notches. Relatively smooth surfaces. (See figs. 2(a) to (c)).	86.0-86.5 (in range of expected values for this material)	45-12
100-Grit diamond ground					
Hand lapped	Free of any marks	High luster typical of polished surfaces			2
Severe 60-grit silicon carbide ground	Fine pin-point "porosity" and longitudinal scratches; no grinding cracks	Waviness ("chatter-marks") with a periodicity of 3/16 in.	Numerous tiny notches. Surfaces are rough in comparison with the ground or lapped surfaces above. (See figs. 2(d) to (f)).		20
Vapor blasted	Fine pin-point "porosity"	Dull surfaces; edges are slightly rounded			65
Grit blasted				84.5	60
Oxidized		Black adherent coating	Outer layer of solid oxide and a second layer of oxide penetration. Each layer is about 0.0010 to 0.0015 in. thick. Highly irregular oxide-cermet interface containing many large notches. No apparent change in microstructure in the zone below the oxidized layers after 100 hr at 1800° F. (See fig. 2(g)).	(c)	435
Oxidized and 220-grit diamond ground		Similar to 220-grit diamond ground	Surfaces varied from a mixture of oxide and cermet (such as is shown in the partially oxidized second layer of fig. 2(g)) to no evidence of oxide.	85.5	12
Oxidized and grit blasted		Similar to grit blasted		85.0	60
Acid roughened		Gray powdery deposit on surface; several smooth grooves	Highly irregular surface containing many large notches. A layer of weakly bonded carbide particles is at the surface because of preferential attack of the binder phase by the acid. (See fig. 2(h)).	(c)	45

^aFor traverses parallel and perpendicular to the specimen axis the values were, respectively, 6 and 12 rms microin.

^bThe oxides identified from an X-ray diffraction pattern of the surface of the oxidized specimen and the ASTM card index of diffraction data are TiO_2 , Fe_2O_3 , and NiO . The presence of iron oxide is verified by chemical analyses, which indicate that iron is generally present as an impurity to the extent of 1 to 3 percent. The iron is probably picked up during the ball milling of the powders.

^cLow hardness probably due to inhomogeneous and defective surface layers. This value does not reflect the true hardness of the cermet.

^dThe oxide-cermet interface is significantly rougher than the surface. The value in the table is from the surface measurement.

TABLE III. - STRENGTH PROPERTIES OF SURFACE-
TREATED TITANIUM CARBIDE - NICKEL
BASE CERMET (K152B)

Surface treatment ^a	Modulus of rupture, psi	Impact strength, in.-lb
As-ground	202,000	3.8
220-grit diamond (I-A)	191,000	2.4
As-ground	194,000	4.1
100-grit diamond (II-B)	174,000	3.3
Lapped (III-A)	179,000	3.9
	179,000	3.3
	178,000	3.1
	146,000	3.1
Severe silicon car- bide abrasive ground (IV-A)	84,000	3.3
	80,000	2.7
Vapor blasted (V-A)	199,000	3.7
	182,000	3.4
Grit blasted (VI-A)	202,000	3.3
	202,000	2.6
Oxidized (1600° F; 100 hr) (VII-A,B)	^b 147,000	^b 1.8
	^b 147,000	^b 1.6
	^c 146,000	^c 1.2
	^c 139,000	^c 1.2
Oxidized and diamond abrasive ground (VIII-A)	183,000	
	176,000	
Oxidized and grit blasted (IX-A)	200,000	
	168,000	
Acid roughened ^a (X-A)	165,000	0.9
	158,000	.8

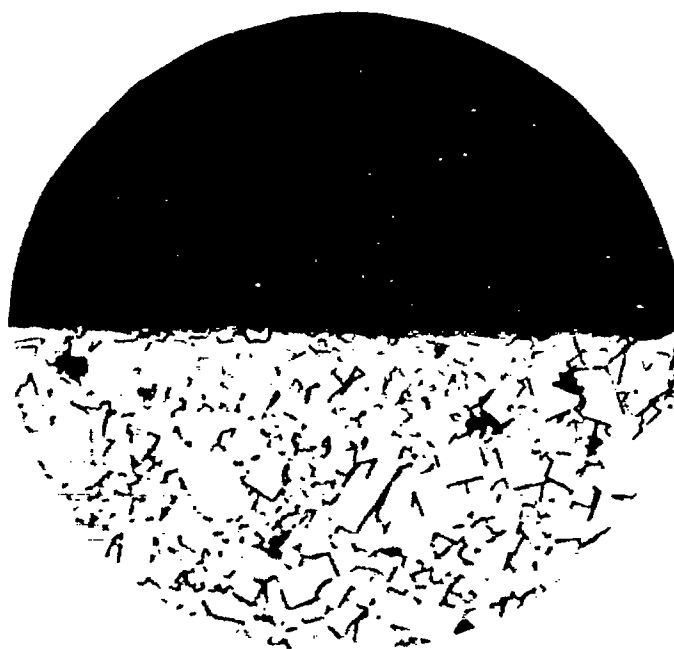
^aCross-sectional dimensions of test specimens were
undersize (0.165 instead of 0.188 in. sq).

^bTreatment VII-B.

^cTreatment VII-A.

4079

CG-2 back



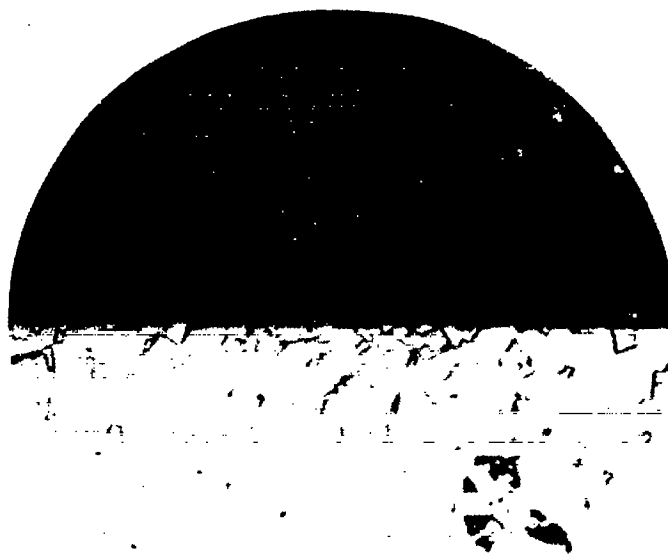
(a) 220-Grit diamond-abrasive ground.



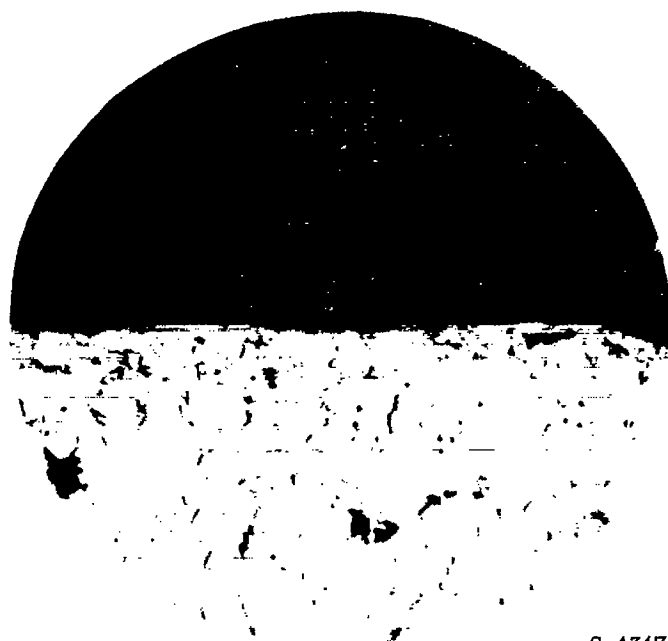
C-43471

(b) 100-Grit diamond-abrasive ground.

Figure 1. - Microstructure of surface-treated specimens. No etch; X1000.



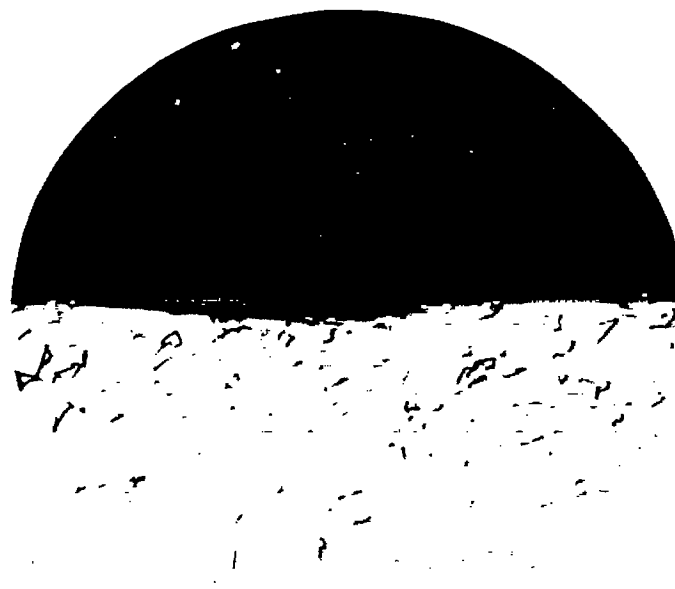
(c) Diamond lapped.



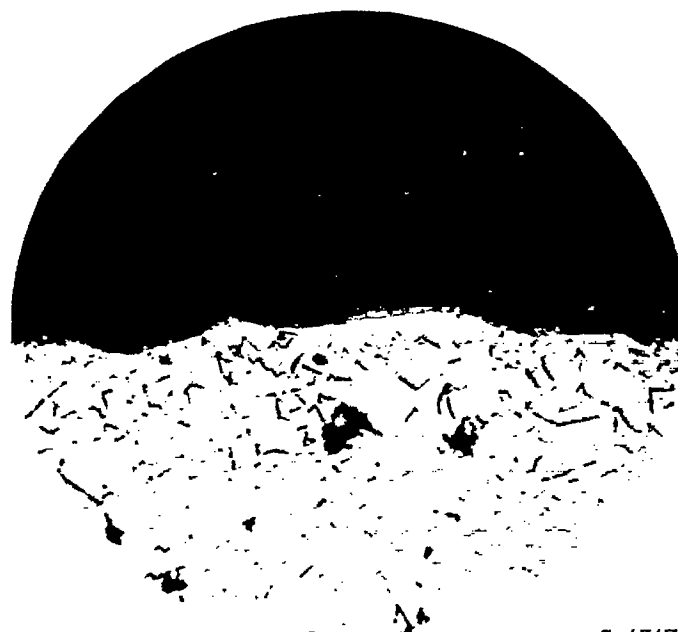
C-43472

(d) Severe silicon carbide abrasive ground.

Figure 1. - Continued. Microstructure of surface-treated specimens. No etch; X1000.



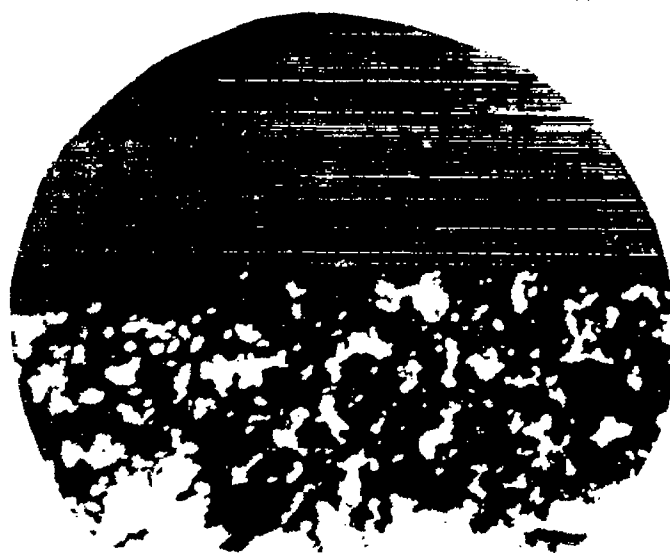
(e) Vapor blasted.



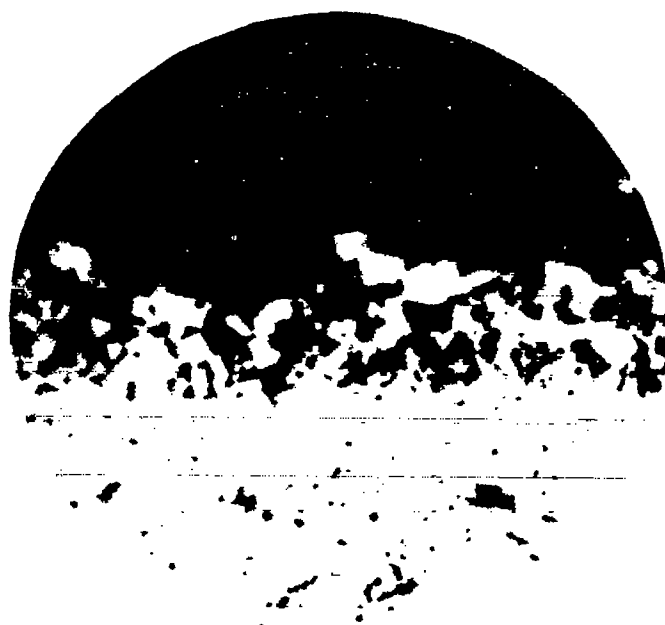
C-43473

(f) Steel grit blasted.

Figure 1. - Continued. Microstructure of surface-treated specimens. No etch; X1000.



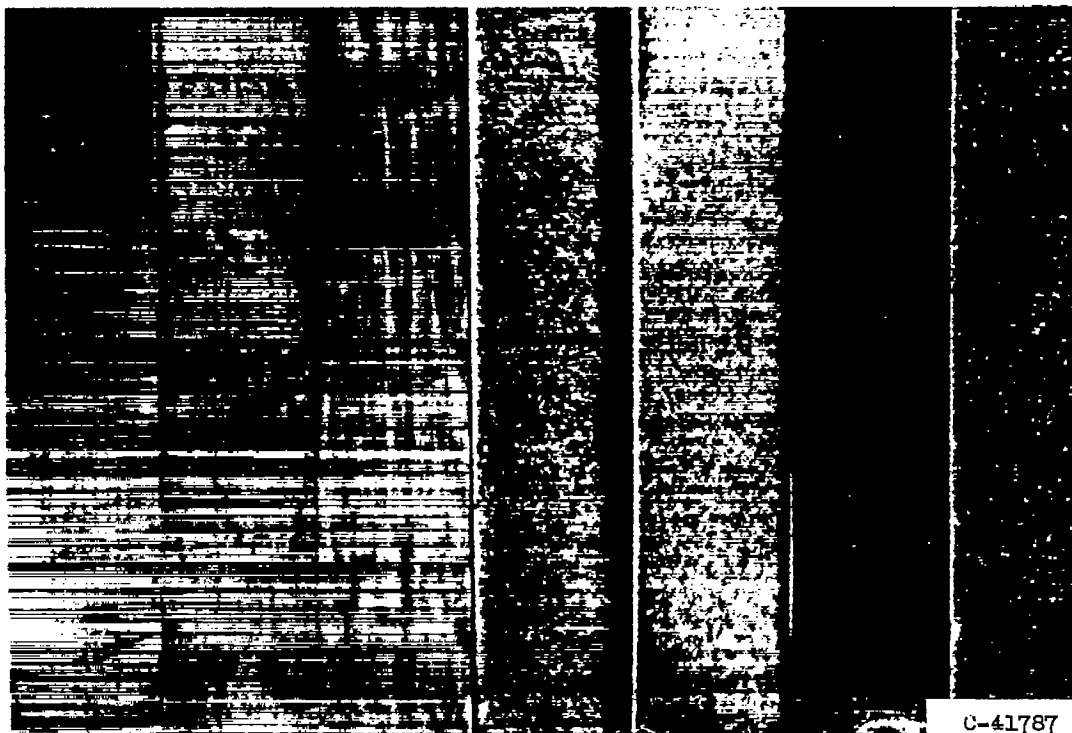
(g) Oxidized for 100 hours at 1600° F.



C-43474

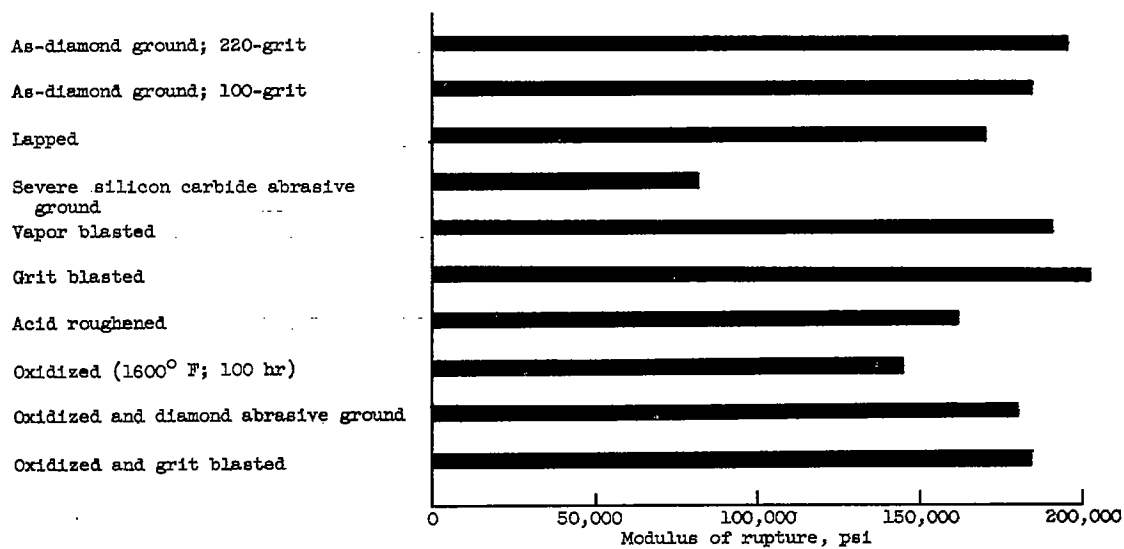
(h) Roughened with aqua regia.

Figure 1. - Concluded. Microstructure of surface-treated specimens. No etch; X1000.

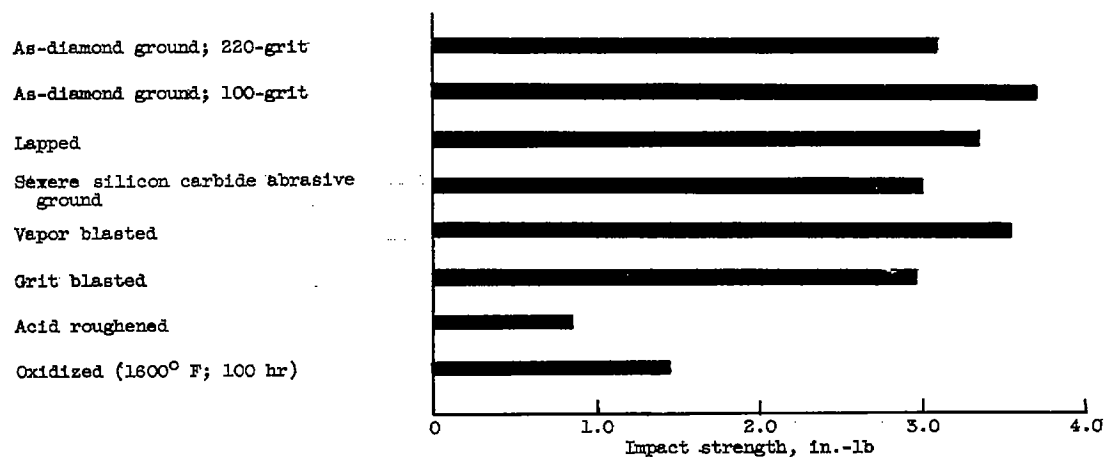


- | | | | | | | |
|---------------------------------|--|---|------------------------------|------------------------------|---------------------------|-------------------------------|
| (a) Diamond
lapped
(III). | (b) Diamond-
abrasive
ground
(I). | (c) Severe
silicon
carbide
abrasive
ground
(IV). | (d) Grit
blasted
(VI). | (e) Vapor
blasted
(V). | (f) Oxidized
(VII(A)). | (g) Acid
roughened
(X). |
|---------------------------------|--|---|------------------------------|------------------------------|---------------------------|-------------------------------|

Figure 2. - Surface-treated test specimens; X5. (Treatment numbers refer to table I.)



(a) Average modulus of rupture.



(b) Average impact strength.

Figure 3. - Effect of surface treatment on the strength of titanium carbide - nickel base cermet (KL52B).